ULTRASONIC SENSING OF POROUS GRANULAR MEDIA

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INTRODUCTION

Emerging high temperature materials such as intermetallic alloys and composites are intrinsically brittle and cannot be either processed or machined by conventional methods. Near net shape processing (of rapidly solidified powders and plasma sprayed foils) using hot isostatic or vacuum hot pressing has recently emerged as a promising method for overcoming these problems. Interestingly, these consolidation processes determine both the component's final shape and its mechanical properties (which depend on relative density, grain size, etc.). Thus a need has emerged for the control of mechanical properties (1,2).

Conventional control of hot isostatic or vacuum hot pressing involves the sensing and feedback of temperature and pressure so as to maintain process variables (temperature and pressure) near previously determined fixed points. Schedules for both variables are determined by trial and error; a process that may take many months or years of testing. New control strategies are seeking to shift the focus of sensing and control to achievement of a goal state combination of properties and component shapes (1,2). While product properties cannot usually be directly sensed during processing, they may sometimes be inferred by sensing microstructure and using previously established relationships between microstructure and properties to indirectly control mechanical properties (3).

The new control approaches for hot isostatic pressing utilize advanced sensors to measure (and control) not only temperature and pressure but also the properties that determine the component's fitness for its intended use (e.g. density and grain size). Predictive models that relate these quantities to the process variables (pressure and temperature) are used to determine the correction to the T,P schedule necessary to affect control of the component properties. Constrained optimization methods can then be used to resolve the conflicting needs of an optimal schedule to achieve full densification with acceptible grain growth (4). The work reported here is concerned with the development of advanced sensing techniques based on emerging NDE methods to measure, during processing, the microstructural state. In our work we are investigating ultrasonic techniques with a view to the development

Review of Progress in Quantitative Nondestructive Evaluation, Vol. 10B
Edited by D.O. Thompson and D.E. Chimenti, Plenum Press, New York, 1991

of in-situ sensors for in-process monitoring of porosity and grain coarsening. These studies extend the work of several researchers who have also investigated ultrasonic techniques for microstructural characterization in powder metallurgical materials (5,13,14).

POROSITY SENSING

The strength and moduli of parts produced by powder consolidation processes are a very strong function of the density. It has been shown (6,7) that the strength depends not only on porosity but also on the spatial distribution and the shape of the pores (7). Porosity can be sensed by a variety of methods including X-ray radiography, eddy currents and ultrasonics. Here, we report on the feasibility of an ultrasonic velocity for determining the pore content.

Measurements

Our research was performed on aluminum powder (nominally 99.5% pure) samples which, for these feasibility studies, had been subjected to confined compression. The samples were 2.54 cm in diameter and ranged in thickness from about 10 mm to 15 mm. The densities of the specimens were determined (to ±2%) by accurate weight and volume measurements and ranged from less than 1.9 gm/cc to 2.7 gm/cc (i.e. porosities ranging from 30% to 1%). The ultrasonic measurements were made using a tone burst ultrasonic system using broadband, highly damped longitudinal and shear wave piezoelectric transducers. Both time of flight and attenuation measurements were made. Transit time measurements were reproducible to 10 nsec and the attenuation difference between acoustic echoes could be measured to 0.001dB. However sample to sample variations were much greater due to irreproducible acoustic coupling and we believe the velocity measurement has a relative 0.1% error and the attenuation 0.05dB.

The ultrasonic phase velocity for both longitudinal and shear waves was measured for all samples. Figs. 1a and 1b show the results for longitudinal and shear wave velocities with transducers with center frequencies of 2.25 MHz and 5.00 MHz. A least squares fit to the data is shown. We see that the velocity was a strong function of the density (and hence the porosity), and the dependence, at this level of measurement precison, was relatively unaffected by frequency of measurement. It can be seen that a measurement of velocity can be used to infer density provided one has a calibration curve such as shown in Fig. 1 or a good theory. The data indicate that the density can be determined to ±2% with p-waves and a little better with s-waves.

Modelling

When a wave of radial frequency ω ($=2\pi f$) propagates through a porous material, the wavenumber is complex i.e. $ß=k+i\alpha$. Thus, there is both a change of phase velocity (ω/k) (where k is the wavenumber) and attenuation (α) when there exists a non zero concentration of pores.

The propagation and scattering of ultrasound in porous media has been widely studied (8-10). We have compared our experimental results with the velocity predictions of the models of Ying, Truell and Waterman (8,9) and Sayers and Smith (10). All the models assume that the scatterers (i.e. pores) are spherical in shape and are of a constant size. To compare with our data, we have assumed the pore size increases linearly with decreasing density (Δ). The pore radius, a, has been assumed zero in the fully dense sample and 150 μm in the sample whose density is 70% of the theoretical density.

The number of scatterers per unit volume, n_0, is then $(1-\Delta)\left(\frac{4}{3}\pi a^3\right)^{-1}$.

In the multiple scattering model of Waterman and Truell (8), the longitudinal wavenumber β_L of the porous body is given by:

$$\left(\frac{\beta_L}{k_L}\right)^2 = 1+\frac{4\pi n_0}{k_L^2}f(0)+\frac{4\pi^2 n_0^2}{k_L^4}\left[f^2(0)-f^2(\pi)\right] \qquad (1)$$

where k_L is the longitudinal wavenumber of the matrix material, n_0 is the number of pores per unit volume, $n_0=(1-\Delta)\left(\frac{4}{3}\pi a^3\right)^{-1}$, $f(0)$ and $f(\pi)$ are the forward and the backward longitudinal scattering amplitudes, respectively. Neglecting the multiple scattering effect, e.q. (1) becomes:

$$\left(\frac{\beta_L}{k_L}\right)^2 = 1+\frac{4\pi n_0}{k_L^2}f(0) \qquad (2)$$

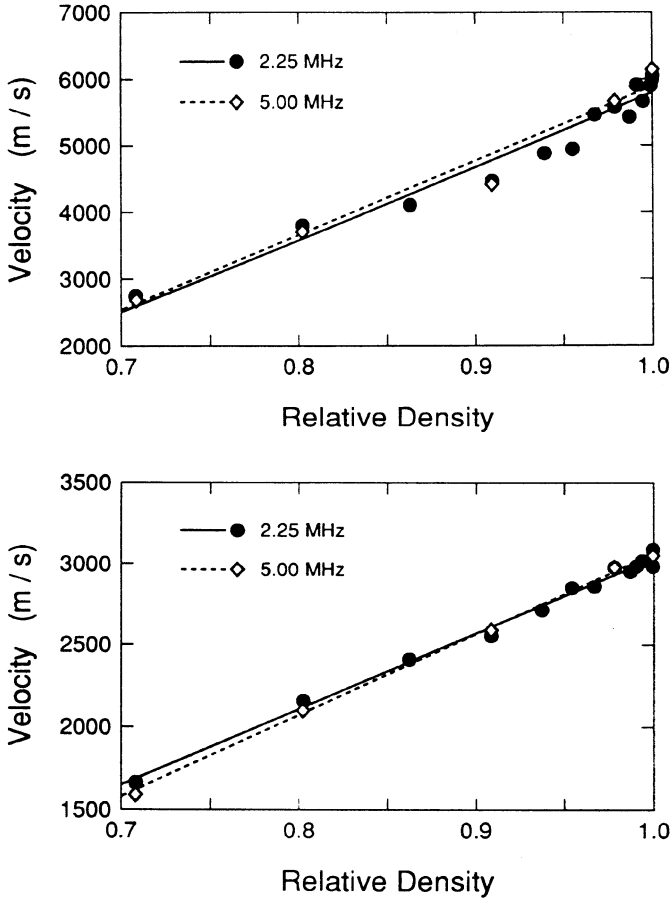

Figure 1. Ultrasonic velocity variation with density. (a) Longitudinal and (b) shear wave data.

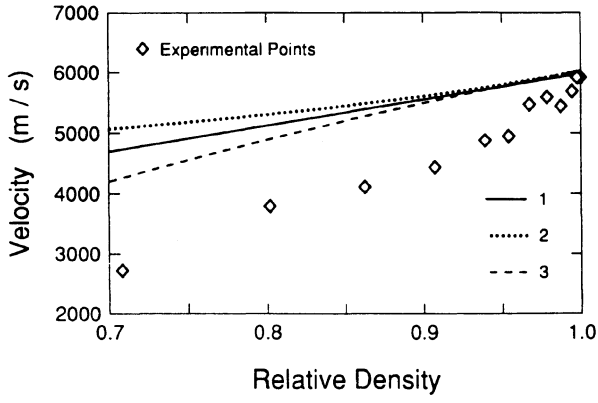

Figure 2. Longitudinal wave velocity - Theory and experiment. (1) Independent scattering model, (2) Multiple scattering, and (3) Self consistent model.

In the so called self-consistent model of Sayers and Smith (10), the effective phase velocities are found by solving:

$$2V_{te}^4 + \left[(6\delta - 5) + \frac{3}{4}(3-\delta)\kappa^2\right]\frac{V_t^2}{1-\delta}V_{te}^2 + 3\frac{1-2\delta}{(1-\delta)^2}\left(1 - \frac{3}{4}\kappa^2\right)V_t^4 = 0 \tag{3}$$

$$V_{le}^2 = V_{te}^2 \frac{\frac{4}{3}\left[(1-P) + \frac{3}{4}P\kappa^2\right]}{\left(1 - P\delta + \frac{3}{4}P\delta\kappa^2\right)} \tag{4}$$

where V_l, V_t are longitudinal and shear wave velocities of the matrix, δ is the porosity (=1-Δ), $\kappa = V_l/V_t$, V_{le}, V_{te} are the effective longitudinal and shear wave velocities of the porous body and $P=\mu/\mu_e$, μ being the shear modulus in matrix and μ_e the effective shear modulus of the porous body.

When we compare the predictions of these three models with our data, Fig. 2, we find that there exists a significant discrepancy between the theoretical predictions and the experimental observations. Since our experimental data was reasonably close to that obtained by Sachse and his coworkers on similar material (13), and the experimental uncertainty is small we suspect the models do not approximate these samples sufficiently well. The disagreement between theory and experiment could be attributed to the two principle assumptions of existing models i.e. spherical scatterers of uniform size. In reality the scatterers are both of varying shapes and sizes. The predominantly uniaxial deformation used flattens pores, and results in a wide range of sizes. Both effects can significantly affect the ultrasonic velocity. Clearly there is a need to better model the scattering for the less ideal pore populations of engineering materials if one seeks to utilize these models to interpret ultrasonic data.

GRAIN SIZE SENSING

Measurements

Ultrasonic wave attenuation is being explored as a means to determine the grain size. Three sets of samples were prepared by

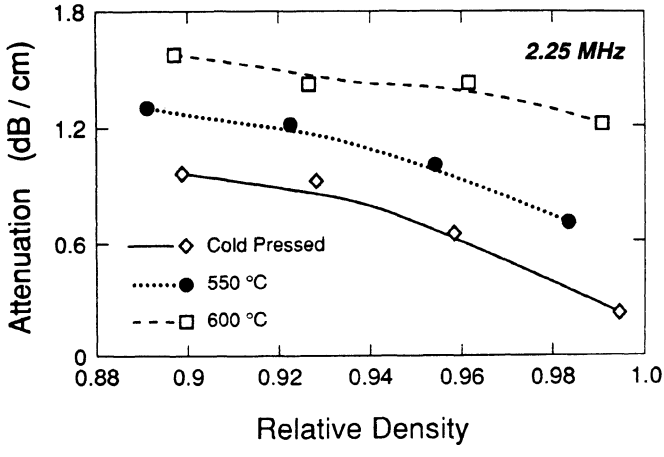

Figure 3. Variation of longitudinal wave attenuation with density and annealing temperatures.

cold compacting aluminum powder. One set was tested with the as received grain size (20μm) while the other two were annealed (at 550°C and 600°C) for one hour resulting in grain sizes of 48 and 71μm. Ultrasonic attenuation measurements were made on all three sample sets. Fig. 3 shows the variation of attenuation with density using a 2.25 MHz p-wave transducer. The measurements were corrected for diffraction (11), and bond losses. The bond loss effect was taken into account by assuming that the couplant had the acoustical properties of a water film 20μm in thickness. From Fig. 3 it is clear that grain growth has a very significant effect upon the ultrasonic wave attenuation. But it is also clear that there exists a contribution from the pores, which decreases with relative density.

In porous granular media the ultrasonic attenuation can be attributed to a combination of scattering from pores and the grains.

$$\alpha = \alpha_{pore} + \alpha_{grain} \quad (5)$$

where α_{pore} is the attenuation due to the pore scattering, α_{grain} is the attenuation due to grain scattering and α is the total attenuation. If the pore concentration is known, then the contribution of the pores to the scattering (α_{pore}) can be deduced either from a calibration curve for samples of small grain size or the predictions of a model. Then, the grain size could be deduced from α_{grain}, again either from a calibration curve or from the predictions of a model (e.g. ref. 12)

Figure 4 shows that annealing (i.e. increasing the grain size) at this level of measurement precision did not have a significant effect on the wave velocity in the samples. Thus, a velocity measurement, and a calibration curve such as Figure 1, appears a good method for inferring the pore concentration.

Modelling

First, consider the contribution to the attenuation from pore scattering (α_{pore}). Attenuation caused by the porosity can be determined by a multiple scattering theory (9). The complex wavenumber is given by eq. (1). At low frequencies it can be

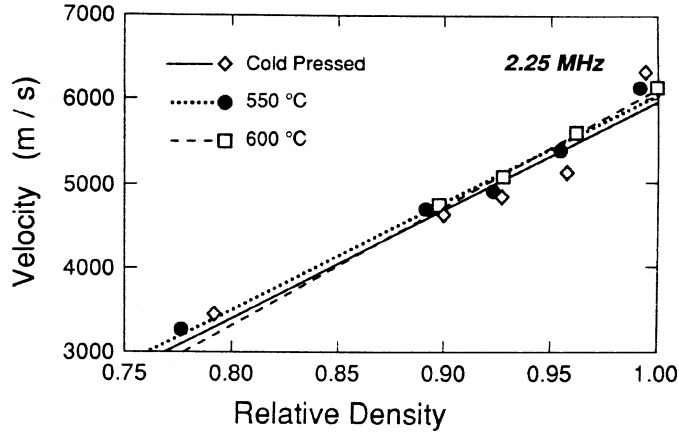

Figure 4. Variation of longitudinal wave velocity with density and annealing temperatures.

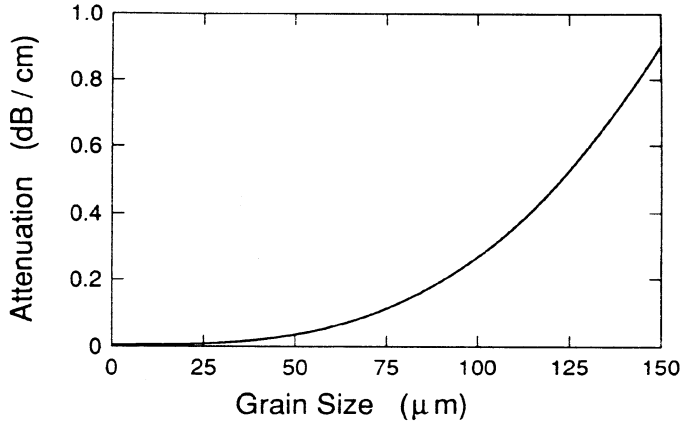

Figure 5. Predicted variation in attenuation due to scattering from grain boundaries with grain size.

simplified to the form:

$$\beta^2 = k_0^2 - \frac{4}{3}\pi a^3 n_0 (C_1 k_0^2 + C_2 k_0^4 a^2 - iC_3 k_0^5 a^3) \qquad (6)$$

Here n_0 is the density of the pores, and a is the pore radius. C_1, C_2 and C_3 are constants. The attenuation coefficient caused by pore scattering is obtained from the imaginary part of ß and can be substracted from the total attenuation. Now the attenuation due to the grains can be calculated from equation (5). At the low frequency or Raleigh limit the attenuation due to the grains can be expressed as:

$$\alpha_{grain} = Tf^4 S \qquad (7)$$

where T is the average grain volume, a measure of the grain size, f is the frequency and S is a material parameter. Elastic wave propagation in granular media has been dealt extensively by Stanke and Kino (12). Based on their model the variation of attenuation

with grain size has been calculated and is shown in Fig. 5. If it is assumed that the grains are of single size, the attenuation predicted by the Stanke and Kino model is found to be smaller than the experimental observations when account is made of the pore scattering contributions. This suggests that for the short term, we should use calibration data to infer grain size for samples of varying density. Further work needs to be devoted toward better understanding and modelling the attenuation and velocity of materials containing broad size and shape distributions of pores and grains.

SUMMARY

This study has demonstrated the existence of phenomena for sensing porosity and grain growth in powder metallurgical processes using ultrasonic techniques. Ultrasonic velocity is a strong function of pore concentration (but depends only weakly on grain size) and provides a potential means for insitu measurement of this quantity. Ultrasonic attenuation, measured in the low megahertz range, depended upon the grain size and the pore content. Using velocity data and a scattering theory to account for the attenuation due to pores, attenuation data may be used to infer the grain size. The predictions of existing scattering theories were compared with the experimental observations. The substantial disagreements between theory and experiment are, we believe, attributable to modelling inadequacies. In particular, the distributions of pore size, pore shape, and grain size that exist in engineering materials need to be better treated in scattering theories.

ACKNOWLEDGEMENTS

We are grateful to T. Watson and J. Wert who provided the samples for this study. The work reported here was funded by the Defense Advanced Research Projects Agency (W. Barker, Program Manager) and the National Aeronautics and Space Administration (Contract Number NAGW-1692).

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